IS 3522-1 (1989): Methods for estimation of common preservatives on textiles - Part 1 [TXD 5: Chemical Methods of Test]

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(Reaffirmed 2005)

Indian Standard

METHODS FOR ESTIMATION OF COMMON PRESERVATIVES ON TEXTILES – PART 1

(First Revision)

भारतीय मानक

वस्त्र में सामान्य परिरक्षी का मूल्यांकन करने की पद्धति – भाग 1

(पहला पुनरीक्षण)

UDC 677·047·62 : 677·016·8

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Price Group 4

FOREWORD

This Indian Standard (Part 1) (First Revision) was adopted by the Bureau of Indian Standards on 31 July 1989, after the draft finalized by the Chemical Methods of Test Sectional Committee had been approved by the Textile Division Council.

This standard was first published in 1966 and has been revised to make it up to date on the basis of experience gained during its use. In this revision the following changes have been carried out:

a) The title and scope have been modified because the methods prescribe the estimation of common preservatives on textiles and not their analysis as such.

b) Conditioning of the samples have been included.

c) Methods B and C given for estimation of salicylanilide have been deleted as these methods are not very accurate and the results may not be comparable with the method specified in this revision.

d) Method for estimation of pentachlorophenol has been replaced by more accurate method based on extraction with 1, 1, 1-trichloroethane and then estimating the pentachlorophenol by spectrophotometer.

e) The estimation of total zinc content in textiles treated with zinc chloride or zinc napthenate, etc, has been given as single method instead of two separate methods.

f) The method for estimation of copper in copper naphthenate treated textiles has been replaced by benzene extraction method because it extracts only the organic copper which is essentially needed for preservation by copper naphthenate. The copper may be adjusted by inorganic salts also, which are not required in the process.

g) The method for estimation of copper-8 quinolinolate has been replaced by a spectrophotometric method for copper 8-hydroxyquniolin.

h) The method for estimation of salicylanilide content in copper salicylanilide treated textiles has been modified for more accurate results.

j) All the mass determinations have been specified according to conditioned mass in the standard atmosphere and results have been expressed on conditioned mass basis.

During storage or in use, most of the textile materials are liable to suffer damage as a result of attack by bacteria, fungi or moulds. Numerous treatments have been developed for textile materials with different preservatives or microbicides, commonly known as fungicides and rotproofing agents, to protect the material from staining and degradation arising from attack and growth of micro-organisms.

The preservatives used for protection of textiles against moulds or fungi are known as fungicides, while those which confer protection against both bacteria and fungi are known as rotproofing agents.

The methods prescribed in this standard are applicable in estimating preservatives when present on the yarns and fabrics of different textile materials. Every precaution should be taken to protect the yarn or fabric, being sampled.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHODS FOR ESTIMATION OF COMMON PRESERVATIVES ON TEXTILES – PART 1

(First Revision)

1 SCOPE

1.1 This standard (Part 1) prescribes methods for estimating the following preservatives on textiles:

Organic Type

a) Salicylanilide
b) Salicylic acid
c) Pentachlorophenol

Inorganic Type

d) Sodium silicoflouride
e) Zinc chloride
f) Zinc naphthenate

Organic metallic

g) Copper napthanate
h) Copper-8 quinolin
j) Copper salicylanilide

2 REFERENCES

2.1 The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
IS 336 : 1973	Ether (*second revision*)
IS 1070 : 1977	Water for general laboratory use (*second revision*)
IS 1745 : 1978	Petroleum hydrocarbon solvents (*second revision*)

3 SAMPLING

3.1 The quantity of textile material of one definite type and quality delivered to a buyer against one despatch note shall constitute a lot.

3.2 Unless otherwise agreed to between the buyer and the seller, the number of bundles or pieces to be selected at random from a lot shall be in accordance with Table 1 or Table 2, respectively.

Table 1 Sample Size for Yarn

(*Clause* 3.2)

Lot Size (Number of Bundles in the Lot)	Sampling Size (Number of Bundles to be Selected)
(1)	(2)
UP to 150	3
151 ,, 300	4
301 ,, 500	5
501 ,, 1 000	7
1 001 ,, 3 000	8
3 001 ,, 10 000	9
10 001 and above	10

Table 2 Sample Size for Fabrics

(*Clause* 3.2)

Lot Size (Number of Pieces in the Lot)	Sample Size (Number of Pieces to be Selected)
(1)	(2)
UP to 100	2
101 ,, 150	3
151 ,, 300	4
301 ,, 500	5
501 ,, 1 000	7

3.3 From each bundle of yarn or piece of fabric selected as in **3.2**, cut out small portions each weighing about 25 g from at least two different parts and mix them. This shall constitute the test sample. While taking the sample, care shall be taken to exclude a sufficient length of yarn or fabric from both the ends.

4 PREPARATION OF THE TEST SPECIMENS

4.1 Cut the test sample into small pieces. Mix all the pieces thoroughly. Draw at least four test specimens from among these pieces each weighing about 5 g or as required in the test.

5 QUALITY OF REAGENTS

5.1 Unless specified otherwise, pure chemicals shall be employed in the tests and distilled water

(*see* IS 1070 : 1977) shall be used where the use of water as reagent is intended.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the test results.

6 CONDITIONING AND TESTING ATMOSPHERE

6.1 All the test specimens prior to test shall be conditioned to moisture equilibrium from the dry side in the standard atmosphere at 65 ± 2 percent relative humidity and 27 ± 2°C temperature, at least for 24 hours. All the mass determinations shall be made in the standard atmosphere after conditioning.

7 ESTIMATION OF SLICYLANILIDE

7.1 Principle

The salicylanilide is extracted from the textile by means of a dilute solution of disodium tetraborate and the extract is treated with a solution of 2, 6-dibromo-p-benzoquinonechlorimine. The blue indophenol colouring matter so formed is absorbed on a disc of Whatman No. 40 filter paper the shade of which is then compared, with the shades of a series of similar discs 'dyed' with the indophenol obtained from solutions containing different known amount of salicylanidide.

7.2 Reagents

7.2.1 *Disodium Tetraborate, 30 g/l Extraction Solution*

Prepared by dissolving 30 g of disodium tetraborate in about 900 ml of water. Add about 30 ml of cyclohexanol then make up to 1 000 ml with water. Shake the solution, remove excess of cyclohexanol by filtration through several layers of filter paper.

7.2.2 *Disodium Tetraborate, 5 g/1, Reagent, Wash Solution*

7.2.3 *2, 6-dibromo-p-benzoquinone Chlorimine 1 g/l Reagent Solution*

The solution is prepared by dissolving 0·1 g in 100 ml of 95 percent V/V ethanol. This solution is unstable; prepare it freshly within a few hours of use.

7.2.4 *Salicylanilide*

0·125 g/1, standard reference solution, prepared by gently heating 0·125 g of pure crystalized salicylanilide in 5 ml of water and 2 ml of 2 M sodium hydroxide solution. When the solid has been dissolved completely dilute to 1 000 ml with water.

7.3 Apparatus

7.3.1 *Paper Discs, 20 mm diameter.*

7.4 Procedure

7.4.1 Place three weighed portions of 0·50 g, 0·25 g, and 0·125 g respectively in separate, dry, flat-bottomed specimen tubes (75 mm × 20 mm) and add to each 5 ml of disodium tetraborate extraction solution. Allow a minimum period of 1/hour for the extraction, which is assisted by occasional gentle shaking or stirring. Extracts containing much suspended matter should be filtered or centrifuged before use but small amounts of suspended fibre are not detrimental. Prepare a series of working standard solutions of salicylanilide by suitable dilution of the reference solution with the disodium tetraborate extraction solution to cover the anticipated salcylanilide content of the material. A suitable range of prepared standard solution can be made as shown in Table 3.

Table 3 Standard Solution of Salicylanilide

(*Clause* 7.4.1)

Disc	Standard Reference Solution	Disodium Tetraborate Extraction Solution	Percentage Salicylanilide on Mass of Material Taken		
			0·5 g	0·25 g	0·125 g
	ml	ml			
1	10·0	15·0	0·05	0·10	0·20
2	9·0	16·0	0·045	0·09	0·18
3	8·0	17·0	0·04	0·08	0·16
4	7·0	18·0	0·035	0·07	0·14
5	6·0	19·0	0·03	0·06	0·12
6	5·0	20·0	0·025	0·05	0·10
7	4·0	21·0	0·02	0·04	0·08
8	3·0	22·0	0·015	0·03	0·06
9	2·0	23·0	0·01	0·02	0·04
10	1·0	24 0	0·005	0·01	0·02
11	0·5	24·5	0·002 5	0·005	0·01

7.4.2 Place 1·0 ml of each extract and of each of the standard solutions in a separate dry specimen tube (100 mm × 25 mm) and mix with 0·2 ml of 2,6-dibromo-p-benzoquinone chlorimine reagent solution. Place immediately a filter paper disc in each tube and allow the whole to stand for 1 hour with occasional stirring, care being taken to keep each disc below the surface of the liquid. Remove the discs from the tubes, rinse them with the disodium tetraborate wash solution, place them on a white opal glass and cover them with separate microscopical cover slips. Estimate the salicylanilide content of the sample under test by comparing the colours of the disc(s) prepared from it with those prepared from the standard solutions. The percentage salicylanilide is that of the solution corresponding to the 'matching' disc.

NOTE — The comparison of the discs is facilitated by placing each on one a separate small piece of white glazed tile.

7.4.3 Modification of the above procedure may sometimes be necessary. For example, if the material contains colouring matter extractable by the disodium tetraborate solution, it may be necessary to make a preliminary extraction of the salicylanilide with an organic solvent or to separate it from the colouring matter in some other way. Such modifications should, however be dictated by circumstances and are beyond the scope of this method.

8 ESTIMATION OF SALICYLIC ACID

8.1 Reagents

8.1.1 *Sodium Hydroxide Solution*, one percent (*m/v*).

8.1.2 *Phospheric Acid Solution*, 5 percent (*m/v*).

8.1.3 *Petroleum Hydrocarbon Solvent*, boiling range 60° to 80°C (*see* IS 1745 : 1978).

8.1.4 *Ether*, (*see* IS 336 : 1973).

8.1.5 *Standard Sodium Hydroxide Solution*, 0·1N.

8.2 Procedure

8.2.1 Take a test specimen of about 5 g weighed accurately to the nearest mg. Boil it in 200 ml of one percent sodium hydroxide solution for one hour. Filter the solution and wash the residue with hot water till free from alkali. Concentrate the filtrate and the washings so collected to about 100 ml. Add phospharic acid solution till the extract is just acidic. Extract the solution with equal volume of petroleum hydrocarbon solvent or a mixture of equal volumes of petroleum hydrocarbon solvent and ether. Evaporate the solvent to get the residue of salicylic acid. Dissolve the residue in warm 15 ml of 95 percent alcohol (previousy neutralized to Phenol Red solution). Add 20 ml of water to this solution and titrate it against 0·1 N sodium hydroxide solution using phenol red as indicator.

8.3 Calculation

8.3.1 Calculate the amount of salicylic acid in the test specimen by the following formula.

$$S = \frac{0{\cdot}0138 \times a \times 100}{M}$$

where

S = the amount of salicylic acid, in percent, by mass;

a = volume in ml, of 0·1 N sodium hydroxide solution; and

M = mass in g, of the test specimen.

NOTE — 1 ml of 0·1 N sodium hydroxide solution is equivalent to 0·013 8 g of salicylic acid.

8.4 Repeat the test with the remaining test specimens and calculate the amount of salicylic acid in each test specimen.

8.5 Calculate the average of the results obtained as in **8.3** and **8.4** and report it as the amount of salicylic acid in the test sample.

9 ESTIMATION OF PENTACHLOROPHENOL

9.1 Principle

The treated textile is steam distilled in the presence of hydrochloric acid and the pentachlorophenol in the distillate extracted with 1, 1, 1-trichloroethane and complexed with copper sulphate-pyridine reagent. The optical density of the complex in 1, 1, 1-trichloroethane is measured on a suitable spectrophotometer at 450 mm.

NOTES

1 Complete removal of pentachlorophenol from some forms of wool is not always possible.

2 The method is applicable to estimation of pentachlorophenol and also to the estimation of pentachlorophenol in the presence of pentachlorophenyl laurate.

9.2 Reagents

9.2.1 *1, 1, 1 trichloroethane*

9.2.2 *Pyridine*

9.2.3 *Sodium Sulphate, Anhydrous*

9.2.4 *Copper Sulphate Reagent Solution 50 g/l*

9.2.5 *Hydrochloric Acid Concentrated* 36% (*m/m*) (*11 M*)

9.2.6 *Pentachlorophenol Standard Reagent*, recrystallized, melting point 188°C *Min.*

9.3 Procedure

9.3.1 Weigh 2·5 ± 0·05 g of the material, cut into small pieces of not more than 5 mm square and place in a 250 ml round bottomed flask (B 24/29) socket. Add 60 ml of water followed by 20 ml hydrochloric acid and a few anti-bumping granules. Fit the flask up for steam distillation and steam distil the contents of the flask ensuring that a constant volume is maintained by applying gentle heat as necessary. Collect 300 ml of distillate in a suitable receiver, paying particular care to prevent loss of pentachlorophenol in the distillate by having adequate cooling. Discontinue the external heating of the flask a few minutes before disconnecting the steam supply. Disconnect the condenser and fit it vertically over the distillate receiver. Wash down the condenser with 25 ml to 30 ml of trichloroethane and collect the washings in the distillate. Transfer the distillate

and trichloroethane washing to 500 ml seperating funnel and shake thoroughly. Allow the layers of water and trichloroethane to separate completely before running off the trichloroethane layer into a 100 ml separating funnel. Wash condenser and distillate receiver with a further 25 ml to 30 ml of trichloroethane and add this to the aqueous solution in the 500 ml seperating funnel.

9.3.2 Repeat the extraction as previously described and add the trichloroethane layer to the first trichloroethane extract in the 100 ml separating funnel. Add to the bulked trichloroethane extract 10 ml of copper sulphate-pyridine reagent (prepared by mixing 4 ml of pyridine with 8 ml of copper sulphate solution immediately before use), and shake well. After effecting complete separation of the aqueous and trichloroethane layers run the lower tricloroethane layer into 100 ml standard flask via a small funnel containing an hydrous sodium sulphate supported by means of a quartz wool plug. Add a small quantity of trichloroethane to the copper sulphate-pyridine solution remaining in the separating funnel, shake and allow the layers to separate before running the trichloroethane layer through the quartz wool filter and collect in the standard flask. Wash the filter with further small quantities of trichloroethane and finally make up to 100 ml with trichloroethane.

9.3.3 Determine the optical density of the solution using a suitable spectrophotometer at a wavelength of 456 nm using trichloroethane as a blank. Estimate the pentachlorophenol content by reference to a calibration graph prepared from known standards of pentachlorophenol.

9.4 Calibration

9.4.1 *Direct*

Prepare a calibration graph using 5, 10 and 15 ml aliquots of a standard solution of pentachlorophenol reagent in trichloroethane (1 g/200 ml) to cover a range of 1, 0, 2·0 and 3·0 percent respectively. Dilute each aliquot to 50 ml to 60 ml with trichloroethane. Add 10 ml of copper sulphate-pyridine reagent and proceed as described in **9.3.1** to **9.3.3**.

Plot optical density against concentration of pentachlorophenol.

9.4.2 *Indirect*

Prepare a calibration graph using 5, 10 and 15 ml aliquots of a standard solution of pentachlorophenol reagent (1 g/200 ml) in dilute sodium hydroxide (only sufficient hydroxide solution to ensure complete solution of the pentachlorophenol is necessary). Place each aliquot in a round bottomed flask, add 60 ml of water and 20 ml hydrochloric acid. Fit the flask for distillation and proceed as described in **9.3.1** to **9.3.3**.

If the distillation technique is satisfactory then the graph obtained by the procedure described under **9.4.1** and **9.4.2** should be the same.

10 ESTIMATION OF SODIUM SILICOFLUORIDE

10.1 Reagents

10.1.1 *Standard Sodium Hydroxide Solution*, 0·01 N.

10.1.2 *Calcium Chloride Solution*, 4 N.

10.2 Procedure

10.2.1 Take a test specimen of about 5 g and weighed accurately to the nearest mg.

10.2.2 Extract the specimen with hot water at 80° to 90°C for 30 minutes keeping the material to liquor ratio as 1 : 30. Cool the solution and add 20 ml of calcium chloride solution. Titrate the extract (without filtering) with 0·01 N standard sodium hydroxide using phenolphthalein as indicator.

10.3 Calculation

Calculate the amount of sodium silicofluoride in the test specimen by the following formula:

$$S = \frac{V \times 0{\cdot}000\,47 \times 100}{M}$$

where

S = amount of sodium silicofluoride, percent, by mass;

V = volume, in ml, of 0·01 N sodium hydroxide; and

M = mass in g, of the test specimen.

NOTE — One mm of 0·01 N sodium hydroxide = 0·000 47 g of sodium silicofluoride.

10.4 Repeat the test specimens and calculate the amount of sodium silicofluoride in each test specimen.

10.5 Calculate the average of the results obtained as in **10.3** and **10.4** and report it as the amount of sodium silicofluoride in the test sample.

11 ESTIMATION OF ZINC IN TEXTILES TREATED WITH ZINC CHLORIDE AND ZINC NAPHTHENATE

11.1 General

If this method is used, then jute and similar materials, which contain metal impurities forming oxinates at *p*H to *p*H 6 will be determined as zinc and it is necessary, therefore, to carry out a blank determination on the unproofed material.

NOTE – Where the estimation of zinc relates solely to assessing the content of zinc naphthenate, it is expected that the proofer or finisher will guarantee that the zinc is present solely as naphthenate and is not mixed with other zinc soaps or compounds.

11.2 Volumetric Method

The material is subjected to wet oxidation. The zinc is precipitated as zinc oxinate from a buffered solution, the precipitate dissolved in acid and the oxine content determined after bromination by an iodometric titration.

11.2.1 *Reagents*

11.2.1.1 *Sodium acetate solution M*

11.2.1.2 *Acetic acid 1 M — dilute 5 M reagent solution five times*

11.2.1.3 *Ammonia solution, 5 M reagent solution*

11.2.1.4 *Ammonium chloride, 2 M reagent solution*

11.2.1.5 *8-hydroxyquinolin 20 g/l reagent solution*

The solution is prepared by dissolving 2 g of 8-hydroxyquinolin in 100 ml of M acetic acid.

11.2.1.6 *Hydrochloric acid 2 M, dilute 5 M reagent solution*

11.2.1.7 *Nitric acid, concentrated 70 percent (m/m) (16 M)*

11.2.1.8 *Sulphuric acid, concentrated, 98 percent (m/m) (approximately 18 M).*

11.2.1.9 *Potassium bromate 0·02 M standard volumetric solution*

The solution is prepared by dissolving 2·784 g potassium bromate and 12·0 g potassium bromide in water and making up to 1 000 ml with water.

11.2.1.10 *Sodium thiosulphate 0·1 M standard volumetric solution*

11.2.1.11 *Potassium iodide 0·5 M non-standardized volumetric solution*

11.2.1.12 *Methyl red 0·05 g/l indicator solution*

The solution is prepared by dissolving 0·05 g of methyl red in 1 000 ml of a solution consisting of 800 ml 95 percent (*v/v*) ethanol and 200 ml of water.

11.2.1.13 *Soluble starch indicator solution 10 g/l*

11.2.2 *Procedure*

Weigh accurately about 2 g of the material, transfer to a 200 ml kjeldahl flask. Add 10 ml of sulphuric acid followed by gradual addition of nitric acid until there is no reaction on further addition of acid (this volume can very and may be as much as 30 ml). Apply heat gradually and digest with the further addition of nitric acid if necessary until organic matter is completely destroyed. Evaporate down to fuming, cool, add 5 ml to 10 ml of water and boil to remove nitric acid. Cool, dilute to approximately 50 ml with water and filter if necessary through a Whatman No. 42 filter paper.

11.2.3 To the solution add a few drops of methyl red, make just alkaline with ammonia solution, add 120 ml of ammonium chloride solution, 25 ml of sodium acetate solution, 6 ml of acetic acid and dilute the solution to approximately 200 ml with water. Adjust the *p*H value of the solution approximately to 5·3 by adding acetic acid or ammonia solution as appropriate using a *p*H meter. Heat the solution to 70°C, add 10 ml of 8-hydroxyquinolin reagent solution, bring to the boil and place on a heated steam bath for 30 minutes. Check that there is excess of reagent solution then filter through a suitable sintered glass crucible (e.g. G 3 or G 4) and wash the precipitate with hot water to remove excess 8-hydroxyquinolin i.e. until the washings are free from colour. Transfer the precipitate to a beaker, and extract the crucible with hot hydrochloric acid, then transfer the washings to the beaker, bulk up to approximately 100 ml with hydrochloric acid and boil solution to dissolve the precipitate. Transfer the solution to a 500 ml conical flask. cool, add 1 ml of methyl red indicator solution and titrate with potassium bromate until the colour of the solution becomes sulphur yellow. Add a further 1 ml of indicator solution.

Continue the process until a 1 ml portion of the indicator is decolorized immediately. This second stage of the titration should not take more than a few mm of the potassium bromate solution. Note the volume of titrant added. Allow the solution to stand for 2 minutes to ensure complete bromination of the 8-hydroxyquinolin, add 5 ml of the potassium iodide solution using soluble starch as indicator. The back titration should not exceed 2 ml. Carry out a reagent blank determination using the same amount of indicator solution as used in the determination.

1 ml of 0·2 M potassium bromate solution = 0·000 980 6 g zinc.

12 ESTIMATION OF COPPER IN TEXTILES TREATED WITH COPPER NAPHTHENATE

12.1 General

This method gives the quantity of organic copper as well as nepthanic acids derived from copper naphthenate Use of inorganic copper such as copper sulphate is not recommended in preservative formulations and hence the extraction with benzene has been specified which will extract only

organic copper as naphthenate from which both copper and naphthanic acid can be estimated. In absence of standard naphthenic acids, these may be estimated gravimetrically and copper by colorimetric method.

12.2 Reagents

12.2.1 *Benzene or Petroleum ether G.p, =40° – 60°C)*

12.2.2 *Hydrochloric acid (approx. 6 N)*

12.2.3 *Conc Nitric acid*

12.2.4 *Ammonia solution — 5 M reagent solution*

12.2.5 *Standard Copper solution — prepared as follows:*

"Dissolve 0·983 g Analar grade copper sulphate ($CuSO4.5H_2O$) in distilled water and make up to 500 ml in a volumetric flask.

1 ml of this solution = 0·5 mg copper (as Cu)

Standard copper solution may also be prepared from electrolytic copper.

12.3 Procedure

12.3.1 From the test sample cut out pieces each weighing about 10 g and condition them to moisture equilibrium in the standard atmosphere of 27 ± 2°C temperature and 65 ± 2 percent relative humidity. Saturated sodium nitrite solution may be used for such atmosphere.

12.3.2 From the test specimens weigh about 10 g of the conditioned material correct to the nearest mg and put in a thimble or properly wrap in a filter paper so that any solid matter detached from the fabric does not enter the extraction flask.

12.3.3 Extract the test specimen thoroughly for 3-4 hours with benzene or petroleum ether in a soxhlet extraction apparatus at the rate of 8-10 extractions per hour.

12.3.4 Recover most of the solvent from the extraction flask and evaporate the remaining solvent by heating the extraction flask at 105 ± 3°C in a oven for 20-30 minutes. A control experiment is run simultaneously for estimation of solvent, extracts other than copper naphthenate. Add 100 ml of hydrochloric acid (6 N) and reflux for 30 min when naphthenic acids (brown colour) will separate out and copper will go into acidic aqueous phase as copper salt. Cool the flask and quantitatively transfer the material to a separating funnel. Extract with solvent ether three times (50 ml, 30 ml), combine the ether extracts and wash with distilled water till the wash water is free from acid. Acidic aqueous layer is released from estimation of copper.

12.3.5 *Estimation of Naphtenic Acids*

12.3.5.1 Take the combined ether extracts in a tared flask and remove the solvent. Add 1-2 ml acetone and heat at 105° ± 3°C for 30 minutes. Weigh the flask containing brown viscous mass of naphthenic acids. Subtract the extractible matter obtained in blank. Estimate the percentage of naphthenic acids by the formula:

$$\text{Naphthenic acids, percent} = \frac{m_2 - m_1}{m}$$

where

m_1 = mass of empty flask,

m_2 = mass of flask plus material minus extractibles obtained in the blank; and

m = mass of the specimen taken.

NOTE — The acid value of the material may be checked by dissolving it in neutral in alcohol and titrating with 0·1 N KOH (with phenolphthalein as indicator. The acid value is found to be 178).

12.3.6 *Estimation of copper*

12.3.6.1 Take the acidic aqueous extract in a beaker (250 ml), add 2-3 ml conc nitric acid and carefully heat to reduce the volume to 10-15 ml. Cool, dilute with water (filter, if necessary), add excess ammonia to develop blue colour and make up the volume to 50 ml.

Take 10, 20, 30 and 40 ml of standard copper solutions in separate 50-ml volumetric flasks, develop blue colour by adding excess ammonia and make up the volume of each to 50 ml. Transfer each solution to colorimer tube and take their colorimeter readings against blank set at zero. Prepare a standard curve (colorimeter reading-Vs-copper concentration). Calculate the amount of copper in the experimental solution from its colorimeter reading and the standard curve. In absence of colorimeter the color of the solution can be matched against that of standard copper solution in Nessler tubes.

13 ESTIMATION OF COPPER 8-HYDROXYQUINOLIN

13.1 General

The method is applicable to the determination of copper 8-hydroxyquinolin in textile materials provided that dyestuffs which are soluble in sulphuric acid or dichloromethane are absent. The copper 8-hydroxyquinolin is extracted from the material by hot extraction with sulphuric acid. The acid solution after neutralizing is extracted with dichloromethane and the optical density of dichloremethane solution is measured on a suitable spectrophotometer at 410 nm.

13.2 Reagents

13.2.1 *Dichloromethane*

13.2.2 *Sodium Sulphate Anhydrous*

13.2.3 *Ammonia Solution, 5 M*

13.2.4 *Sulphuric Acid, 2·5 M*

13.2.5 *Copper 8-hydroxyquinolin*

Standard reference reagent, prepared by adding excess of a solution of copper sulphate (60 g/l) to 100 ml solution of 8-hydroxyquinolin (650 g/l) in 95 percent (*v/v*) ethanol. Filter off the precipitated copper 8-hydroxyquinolin on a suitable sintered glass filter, wash with water to remove excess copper sulphate. Air dry at 90°C for 1 hour. The reagent should be bright yellow in colour.

13.2.6 *Copper 8-hydroxyquinolin*

Standard reference solution, prepared by dissolving 1·000 g standard reference reagent in 1 000 ml of dichgloromethane.

13.2.7 *Bromocresol Green Indicator Solution*

0·4 g/1. Warm 0·1 g of bromocresol green with 2·9 ml of 0·05 M sodium hydroxide solution and 5 ml of 95 percent (*v/v*) ethanol; after solution is effected, add 50 ml of 90 percent (*v/v*) ethanol and dilute to 250 ml with water.

13.3 Procedure

Digest on a boiling water bath for 15 minutes 1·0 g of the finely divided material in a 100 ml beaker with 25 ml of sulphuric acid (with dense fabrics it can be advantageous to place the sample and acid in a suitable high speed disintegrator or homogenizer, then transfer the pulp suspension to the beaker). Filter the extract through a quartz pulg into a 400 ml beaker. Repeat the extraction three times with 25 ml quantities of sulphuric acid filtering each extract through the original quartz wool plug. Cool the bulk solution, add bromocresol green indicator and adjust to *p*H 6 ± 1 with ammonia solution added from a burette, cooling the solution from time to time as necessary. (The *p*H adjustment can be done advantageously by the use of a *p*H meter). Transfer the solution to a 500 ml separating funnel and add 20 ml of dichloromethane. Shake the funnel for at least 1 min. Allow the contexts to separate out, run off the dichloromethane layer through anhydrous sodium sulphate supported on a quartz wool plug directly into a 100 ml graduated flask. Repeat the dichloromethane extraction four times with further 10 ml volumes of dichloromethane, filtering each through the same sodium sulphate plug filter. Bulk the dichloromethane extracts to 100 ml and measure the optical density of the solution on a suitable spectrophotometer at a wavelength of 410 nm using 5 mm cells with dichloromethane as a blank. The calculation of copper 8-hydroxyquinolin may be made from a previously prepared calibration graph.

Calculate the copper content as follows:

Percentage copper content = percentage of copper 8-hydroxyquinolin × 0·180 6.

13.4 Calibration

Take 0·0, 5·0, 7·5 and 10·0 ml of the standard reference reagent equivalent to 0·0, 0·5, 0·75, and 1·0 percent respectively. Place in 100 ml flask and dilute each to 100 ml with dichloromethane. Determine the optical density of the solution in 5 mm cells at a wavelength of 410 nm using dichloromethane as a blank. Prepare graph of optical density against percentage of 8-hydroxyquinolin.

14 ESTIMATION OF COPPER SALICYLANILIDE

14.1 Estimation of Copper Content

14.1.1 Take a test specimen of about 10 g weighed accurately. Transfer the test specimen to a 90 ml porcelain crucible. Place the crucible in a muffle furnace and slowly increase the temperature to about 300°C. After the sample is charred remove the crucible from the furnace.

14.1.2 Cool the crucible and moisten the carbonaceous skeleton with 1 ml sulphuric acid. Heat the contents until white fumes cease to volatilize and grey ash remains in the crucible. Digest the residue with 20 ml of distilled water and 5 ml of sulphuric acid by heating nearly the boil for about 5 minutes. Cool the solution and neutralize it with ammonium hydroxide and add 10 ml of ammouium hydroxide in excess. Make up the volume to 250 ml with distilled water.

14.1.3 Carry out a blank by adding 1 ml of sulphuric acid in a crucible and heating it over a low flame until dense white fumes are no longer evolved. Digest the residue with 20 ml of distilled water and 5 ml of sulphuric acid by heating to boil for about 5 minutes. Cool it and neutralize with ammonium hydroxide and add 10 ml of ammonium hydroxide excess. Make up the volume to 250 ml with distilled water.

14.1.4 Take 50 ml of solution obtained as in **14.1.2** in a 100 ml Nessler-tube. Add 1 ml of gun Arabic solution and 10 ml of sodium diethyl dithiocarbamate solution, mix thoroghly and keep it for comparison.

14.1.5 Take 50 ml of blank solution obtained as in **14.1.3** in a 100 ml Nessler tubes. Add to it a

known quantity of standard copper salution from a 10 ml burette. Add 1 ml of gum Arabic solution and 10 ml of sodium diethyl dithiocarbamate solution, mix thoroughly and compare the colour of the solution with the colour of the extract (*see* **14.1.4**).

NOTES

1 The amount of standard copper solution to be added will depend upon the colour produced by the extract. If the colour in the sample tube is too light for a good comparison, the amount of copper present on the basis of 10 g test specimen is below the sensitivity of the method. Best results would be obtained when the aliquot contains copper equivalent to 1 to 5 ml of standard copper solution.

2 If necessary, water may be added to the tube containing extract to make the volume equal to that of the tube containing the standard solution.

14.1.6 *Calculation*

Calculate the copper content of the test specimen by the following formula:

$$C = \frac{A \times 0{\cdot}001 \times 5}{B}$$

where

C = copper content, in percent by mass;

A = volume, in ml, of standard copper solution; and

B = mass, in g, of the test specimen.

14.1.7 Repeat the test with the remaining test specimens and calculate the copper content of each test specimen.

14.1.8 Calculate the average of the results obtained as in **14.1.6** and **14.1.7** and report it as the copper content of the sample.

NOTE — Alternatively the test specimen may be ashed as given in **14.1.9**.

14.1.9 Take a test specimen weighing about 10 g. Weigh it accurately to the nearest mg. Transfer the test specimen to a 90 ml porcelain crucible. Place the crucible on a low flame and protect it from strong drafts. Volatilize the organic matter gently taking care that the material does not burn with a flame. Continue heating till a carbonaceous skeleton is left.

14.2 Estimation of Salicylanilide Content

14.2.1 *Reagents*

14.2.1.1 *Potassium bromate-bromide solution*

The solution 0·1 N, is prepared by dissolving 2·784 g of potassium bromate and 10 g of potassium bromide in sufficient amount of water and making up the volume to one litre.

14.2.1.2 *Hydrochloric acid*, 4 N.

14.2.1.3 *Potassium iodide solution*

The solution 1 N, is prepared by dissolving 166·028 g of potassium iodide in sufficient amount of water and making up to one litre.

14.2.1.4 *Standard sodium thiosulphate solution*, 0·1 N.

NOTE — The normality of sodium thiosulphate should be checked before use.

14.2.1.5 *Starch solution*

The solution is prepared by dissolving 1 g of soluble starch in 100 ml water.

14.2.1.6 *Sodium hydroxide solution*, 5 percent.

14.2.2 *Procedure*

14.2.2.1 Take a test specimen weighing about 10 g. Weigh it accurately to the nearest mg. Put the test specimen in the conical flask and heat it with 100 ml of sodium hydroxide solution for two hours at about 90°C on a water bath. Filter the extract and wash the residue twice with hot water and once with cold water. Collect the washings and the filtrate and make up to 250 ml with distilled water.

14.2.2.2 Take 25 ml of the extract in a 500 ml stoppered flask. Add to it 50 ml of potassium bromate-bromide solution and 30 ml of 4 N hydrochloric acid. Keep the flask in a cold water-bath at 15°C for half an hour with occasional shaking. Add 30 ml of potassium iodide solution to the flask. Keep it for five minutes and titrate the liberated iodine against sodium thiosulphate solution using starch solution as indicator.

14.2.2.3 Carry out a blank simultaneously following the procedure prescribed in **14.2.2.2** but taking 25 ml of water instead of extract.

14.2.3 *Calculation*

Calculate the salicylanilide content in the test specimen by the following formula:

$$S = \frac{(V_1 - V_2) \times 0{\cdot}003\,55 \times 1\,000}{M}$$

where

S = salicylanilide content, percent, by mass;

V_1 = volume, in ml, of 0·1 N sodium thiosulphate required for extract (*see* **14.2.2.3**); and

V_2 = volume, in ml, of 0·1 N sodium thiosulphate required for extract (*see* **14.2.2.2**); and

M = mass, in g, of the test specimen.

14.2.4 Repeat the test with the remaining test specimens and calculate the salicylanilide content in each test specimen.

14.2.5 Calculate the average of the results obtained as in **14.2.3** and **14.2.4** and report it as the salicylanilide content of the test sample.

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